Developing a NIST Reference Material for Trace Explosives Detection

An important tool to protect against terrorist explosives events is the sensitive and accurate measurement of residues transferred by the handling and fabrication of improvised explosives devices. Currently there are no internationally recognized certified reference materials specifically addressing trace explosives analysis. Commercial solutions of high explosive ingredients that are currently available serve only to evaluate an instrument's final detection step. Solutions cannot be used to test and validate the performance of the entire residue detection protocol, including particle collection and transfer of the characteristic compounds into the instrument prior to the measurement. The goal of this project is to develop a NIST Reference Material (RM) 8105 Trace Particulate Explosives, that can be used to test and calibrate both field-deployable detectors, such as those used in transportation security, as well as sophisticated laboratory instruments, such as those used for post-blast forensic investigations.

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The challenge is to create a material that will provide calibrated trace amounts of two compounds (TNT and RDX) that are commonly used in plastic and military explosives in a form that is safe to handle. Using the RM, customers will be able to test and calibrate a wide variety of trace explosives detection equipment.



This work is part of a larger effort by the Department of Homeland Security to promote standardization for critical measurements. RM development is being done in conjunction with the Analytical Microscopy Group of the Surface and Micro-

analysis Division of NIST, who are investigating the properties of explosives residues and testing the performance of field-deployable explosives detection systems. This project builds on our previous research in analytical methods that support the forensic investigation of improvised explosive devices prepared using gunpowder, which resulted in the development of RM 8107 Additives in Smokeless Powder.

In the past year, five candidate materials for the new RM were prepared and evaluated. Candidate materials were selected to provide high thermal stability and appropriate particle size for representing explosives residues. To fabricate the candidates, the active explosive ingredients RDX and TNT were dissolved in an organic solvent and coated onto a series of inert substrates. The solvent was removed under vacuum using a rotary evaporator leaving trace amounts of the explosives coated on the inert substrates.

Once prepared, each candidate material was extracted, and the explosives content determined by liquid chromatography. To investigate the stability of each candidate material upon storage, subsamples were stored at sub-ambient and elevated temperatures and analyzed over a six-month period. Candidate materials were also evaluated using the most commonly deployed detection system for trace explosives detection, ion mobility spectrometry. From these experiments, a single "best candidate" substrate has been selected – non-porous glass beads with a bonded organosilane coating. We are now developing a protocol to prepare the RM candidate in a one kilogram quantity.

RM 8105 Trace Particulate Explosives will provide customers with a well characterized material to strengthen detection technology: from manufacturers of current technology and their users, to developers of next-generation technology.

Manufacturers of detection equipment will be able to test and document the performance of their instrumentation. Buyers of detection equipment, such as the Transportation Security Agency, will have a material that they can use to "benchmark" the sensitivity, calibration, and reliability of equipment from a number of potential vendors prior to procurement for widespread deployment. Researchers developing new measurement technology can use the material to optimize their detection approach. Forensic laboratories may use the material to validate residue collection protocols as well as the analytical extraction and measurement methods used for explosives evaluations. RM 8105 will provide a means of rendering a trace explosives measurement that is "traceable to NIST."

Future Plans: We intend to complete the fabrication of the RM and make the first round of value-assignment measurements in FY06. A workshop on reference materials for explosives measurements is planned. In future years, we may develop a series of reference materials encompassing a wider array of explosive ingredients used in the fabrication of improvised explosive devices.

Publications:

MacCrehan, W.A. and Bedner, M. "NIST Reference Materials for Explosives Analysis," Proceedings of the 8th International Symposium on the Analysis and Detection of Explosives, Public Security and Emergency Preparedness, Ottawa, ON, Canada, 2004.